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NORAIR DIVISION
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**EVALUATION OF "CERAMIC GOLD" COATINGS
ON TITANIUM**

16 February 1959

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1. INTRODUCTION

There is a need to develop low emissivity coatings for application to metals. One primary production application requires a coating having an emissivity below 0.25. This program was initiated to investigate the low emissivity "ceramic gold" coatings for this application.
2. OBJECT

The object of this program was to evaluate a "ceramic gold" film for use as a low emissivity coating for the titanium engine shrouds of the T-38 Talon Trainer.
3. CONCLUSIONS

"Ceramic gold" film appears suitable for use as a heat-reflecting, low-emissivity coating for the titanium engine shrouds of the T-38 aircraft. Laboratory tests show that the film maintains surface emissivities of 0.15 or lower for an extended period of time without signs of deterioration, provided temperatures much above 900 F are not exceeded. The excellent adhesion to titanium and the chemical inertness of the gold film indicate that the coating should require very little maintenance or repair. Engine service tests conducted with a gold coated shroud panel show that the panel remains considerably cooler and radiates less heat than adjacent panels coated with aluminum paint.
4. TEST PROCEDURES
 - 4.1 Materials
 - 4.1.1 Titanium Ti65A test panels, 0.016 by 4.0 by 4.0 inches.
 - 4.1.2 Titanium Ti65A tensile test specimens, Type F2, 0.016 inch thick.
 - 4.1.3 Titanium Ti65A shroud test panel, 0.012 by 12 by 72 inches.
 - 4.1.4 Hanovia Liquid Gold #261, NH, and NW, Engelhard Industries, Inc., Los Angeles, California.
 - 4.1.5 Extra High Heat Res H-170, Spaco, Inc., Cleveland, Ohio.
 - 4.2 Preparation of Specimens
 - 4.2.1 Titanium specimens were cleaned with acetone. Several panels were polished with crocus cloth prior to cleaning.

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4.2 Preparation of Specimens (Continued)

4.2.2 Liquid gold #261, #2, and #3 were sprayed or brushed to a given thickness on individual panels.

4.2.3 Coated panels were air dried and oven dried according to several time and temperature schedules, noted below.

4.2.4 Panels were fired in an electric globar furnace with the door ajar. Various firing schedules were followed to determine which provided the best visible reflectance and adhesion.

4.2.5 Panels were exposed to 900 F \pm 10 for 72 hours prior to testing to simulate service environment.

4.2.6 A T-38 engine shroud panel was sprayed with #2 gold and air dried for 4 hours. It was then fired in an air circulating electric oven from 200 to 900 F in 3 hours.

4.3 Test Methods

Except where indicated, all of the tests were conducted on one coat of #2 gold applied to Ti6Al titanium having an as-received 2D finish.

4.3.1 Emissivity - Test panels prepared in three different ways were measured for emissivity at 200, 500, 800, 900, and 1000 F on the Norair emissimeter.

4.3.2 Service Evaluation - The coated engine shroud panel was positioned in the T-38 tailcone shroud. Temperature measurements on the gold coated panel and adjacent panels coated with Extra High Heat Res H-170 aluminum paint were made during engine tests.

4.3.3 Flexibility - Test panels were conditioned for 2 hours at -40 F \pm 2 and immediately bent over a cold 1/2 inch diameter steel mandrel.

4.3.4 Impact Resistance - A one inch diameter steel ball weighing 66 grams was dropped onto the surface of the coating from a height of 6 feet.

4.3.5 Fluid Resistance - Test panels were immersed for 72 hours in:

- Water at room temperature
- JP-5 fuel (MIL-F-5624C) at room temperature
- Engine oil (MIL-L-7808C) at 300 F \pm 2
- Hydraulic fluid (MIL-D-5606A) at 300 F \pm 2

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4.3 Test Methods (Continued)

- 4.3.6 Salt Spray Resistance - Test panels were exposed to 20 per cent salt spray solution at 95 ± 5 F for 4 months.
- 4.3.7 Temperature Resistance - The maximum time-at-temperature which the coatings would withstand at various temperatures without loss of brightness were determined.
- 4.3.8 Tensile Properties - Tensile tests were conducted on three coated and three uncoated Type P2 specimens.
- 4.3.9 Abrasion Resistance - A Taber Abraser, with CS-17F wheels with a 1000 gram load on each, was used to determine the number of cycles the coatings would withstand before failure.

5. TEST RESULTS

5.1 Emissivity

Emissivity values at temperature noted

	<u>200 F</u>	<u>500 F</u>	<u>800 F</u>	<u>900 F</u>	<u>1000 F</u>
2D finish, one coat KH	0.12	0.13	0.14	0.15	0.19
Slightly polished finish, one coat KH	0.10	0.11	0.13	0.13	0.17
Moderately polished finish, two coats KH	0.08	0.10	0.12	0.12	0.15

- 5.2 Service Evaluation - No apparent surface deterioration after 35 hours of testing. Gold coated panel averaged 330 F during test, while aluminum painted panels averaged 430 F.
- 5.3 Flexibility - No crackling, flaking, or loss of adhesion.
- 5.4 Impact Resistance - No coating damage.
- 5.5 Fluid Resistance - No coating damage or discoloration.
- 5.6 Salt Spray Resistance - No corrosion spots after exposure for 4 months.
- 5.7 Temperature Resistance - Surface dulled after exposure for 2 hours at temperatures above 900 F. Coating will withstand short exposure at 1100 F.

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5.8 Tensile Properties (Average of 3 Specimens)

	Yield Strength at 0.2% Offset psi	Ultimate Strength psi	Elongation in 2 inches %
Uncoated specimens	62,610	77,590	27.0
Gold coated specimens	61,960	77,450	31.5

5.9 Abrasion Resistance - Coating withstood 95 cycles before exposure of base metal.

6. DISCUSSION

6.1 Application of Coatings

6.1.1 "Ceramic gold" films are made by first depositing onto a surface, by spraying or brushing, a layer of an organo-metallic gold salt, dissolved in suitable organic vehicles, and then reducing the salt by heating. The resultant film is 22-karat gold approximately 5×10^{-3} mils thick. The four most critical considerations in applying the gold film are the type of coating used, the coating thickness prior to firing, the firing furnace atmosphere and ventilation, and the firing schedule. The coatings used in this program were fairly low in actual gold content (about 8 to 10 per cent) and therefore needed to be applied in thick coats or more than one coat in order to obtain films approaching zero porosity. Thick coats must be dried and fired carefully to prevent the coating from blistering and flaking off. Applying more than one coat requires additional firings for each coat, which results in film dullness and increased costs. The furnace or oven atmosphere must be kept sufficiently oxidizing to bring about complete burn-out of the organic matter and to reduce the salt to bright metallic gold. In addition, the firing chamber must be well ventilated to remove smoke or fumes resulting from firing and prevent their settling on and clouding the surface of the gold. As expected, heating schedules are very important. In general they are of long duration, especially for heavily applied coats where rapid volatilization of the organic vehicles causes blistering. Preventing the coatings from rupturing is probably the most important reason why slow firing rates are necessary. Another reason is that the proper furnace atmosphere and ventilation necessary for rapid deposition is often difficult to maintain. It is necessary, regardless of the rate of heating, to expose the coatings to elevated temperatures for a certain minimum time to achieve adequate bond between the gold and the base metal.

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6.1 Application of Coatings (Continued)

6.1.2 In this program K4 gold appeared to produce better films than #216 or K4 and was, therefore, the only coating used for test purposes. Spraying gave better results than brushing. The most satisfactory firing schedule for the K4 gold is noted below. Although this firing schedule was closely followed in preparing all test panels, it represents only one of many schedules which could produce satisfactory films.

- a. Air dry at room temperature for 1/2 hour,
- b. Oven dry at 200 F \pm 10 for 1/2 hour,
- c. Fire from 200 to 700 F in 1-1/2 hours, and then rapidly raise the temperature to 900 F \pm 10 for a 15 minute soak, and
- d. Air cool to room temperature.

6.1.3 By using coatings with high gold content, which could be applied thinly, and firing these in well-ventilated oxidizing atmospheres, the firing schedules could be considerably reduced in both time and complexity. Preliminary investigation of coatings containing 15 to 20 per cent gold, recently made available by the manufacturer, indicates that satisfactory films can be deposited from single coats in less than 1 hour.

6.2 Emissivity Measurements

The emissivity values in paragraph 5.1 show that emissivities from 0.10 to 0.15 were obtained at temperatures up to 900 F. As reported in previous investigations, "ceramic gold" films possess some porosity and as a result the base metal becomes oxidized at elevated temperatures. This accounts for the rapid increase in emissivity that occurs between 900 and 1000 F. The emissivity remains permanently high even at low temperatures because of the irreversibility of the oxidation reaction. It is believed that this emissivity rise at temperatures above 900 F can be reduced by oxidizing the surface prior to coating. Pre-oxidizing the surface should render the titanium less active at elevated temperature and therefore eliminate much of its growth due to oxidation, which appears to disrupt the gold films. When applied on ceramic enamels, which are relatively nonreactive with oxygen at elevated temperature, gold films remain low heat emitters at temperatures well over 1000 F. Another method for reducing the emissivity is by using the new coatings of higher gold content, since they are expected to give less porous coatings. It is important to note the significant effect that surface polish has on emissivity. Lower emissivity values than those reported here could be obtained with more highly polished surfaces.

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6.3 Service Evaluation

6.3.1 Although there have been only 35 hours of service testing on the gold coated shroud panels, the results are extremely promising. Periodic visual inspections of the gold coated panel indicate that no surface deterioration is occurring. The attached thermocouples indicated that the temperature of the gold coated panel (330 F) was approximately 25 per cent less than that of adjacent panels (430 F) coated with aluminum paint. These results were obtained with the engine at idle power, and with a considerable amount of heat being transferred to the gold coated panel by both conduction and convection. Tests conducted with the engine at full power, and with heat transferred primarily by radiation, should produce an even greater difference in temperature.

6.3.2 According to analytical calculations made by Fluid Dynamics from emissivity values previously reported, the expected shroud temperatures on the T-33A aircraft with engine at full power and complete with afterburner should be 1060 F with aluminum paint on both sides of the shroud and inside the outer skin, or 825 F with gold on both sides of the shroud and aluminum paint inside the outer skin. This indicates that there would be considerable advantage realized by gold coating the shrouds.

6.4 Mechanical and Chemical Properties

The test results show that the gold coating has excellent mechanical and chemical properties. Photomicrographs taken of the gold-titanium interface indicate that a strong mechanical bond exists between the two. This is evidenced by the good adhesion shown by flexibility and impact resistance tests. The inherent chemical inertness of gold is responsible for the excellent fluid and salt spray resistance of the film. According to tensile test results, coating titanium with gold has no apparent deleterious effect on the base metal. The intrinsic softness and malleability of gold is responsible for its normally poor abrasion resistance; however, as a thin film it has more than fair resistance to abrasion. The film apparently assumes some of the characteristics of the underlying titanium.

6.5 Weight

It is significant that, because of the extreme thinness of the gold film, approximately 5 millionths of an inch, any increase in weight caused by coating parts with gold would be negligible.